TRANSESTERIFICATION OF JATROPHA CURCAS OIL USING FLY ASH BASED CATALYST TO PRODUCE BIODIESEL

Compiled as a Requirement to Obtain Bachelor Degree in Chemical Engineering
Muhammadiyah University of Surakarta

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2017

APPROVAL PAGE

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MUHAMMADIYAH UNIVERSITY OF SURAKARTA

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Transesterification of Jatropha curcas Oil Catalyst by Solid Base Fly Ash to Produce Biodiesel

MUHAMMADIYAH UNIVERSITY OF SURAKARTA

Abstrak

Penipisan cadangan minyak dan pencemaran lingkungan merupakan isu global yang menyulitkan manusia dalam beberapa dekade ini. Hal ini telah mempengaruhi perekonomian dunia saat ini, terutama negara berkembang seperti Indonesia. Kenaikan harga BBM berdampak langsung pada kenaikan biaya transportasi, biaya produksi industri dan pembangkit tenaga listrik. Ini berarti harus ada sumber energi alternatif. Biodiesel telah diidentifikasi sebagai salah satu pilihan untuk melengkapi bahan bakar konvensional. Produksinya dari sumber biologis terbarukan seperti minyak nabati dan lemak telah ditinjau secara luas. Keunggulannya atas diesel petroleum: aman, terbarukan, tidak beracun, dan mudah terurai, tidak mengandung belerang; dan pelumas yang lebih baik. Penggunaan bahan baku *fly ash* sebagai katalis heterogen untuk transesterifikasi akan memungkinkan membuat *fly ash* menjadi ramah lingkungan sehingga membuat proses lebih ekonomis. Pada penelitian ini, *fly ash* padat dibuat dan diolah menggunakan KOH untuk menjadi katalis basa. Kemudian proses transesterifikasi dilakukan dengan mencampurkan minyak jarak pagar, katalis, dan metanol menjadi metil ester. Penelitian ini mendapatkan hasil pengaruh suhu terhadap hasil (%) produksi biodiesel. Suhu yang lebih tinggi membuat yield (%) meningkat.

Kata kunci: Biodiesel, fly ash, Transesterifikasi, Jatropha curcas

Abstracts

Depletion of oil reserves and environmental pollution is a global issue that is troubling humans within a few decades. This has resulted in soaring oil prices that will have great impact on the economy of the world today, at least developing countries like Indonesia. The increase in fuel prices had a direct impact on the increase in transport costs, the cost of industrial production and power generation. This means that there should be alternative energy sources. Biodiesel has been identified as one of the notable options for at least complementing conventional fuels. Its production from renewable biological sources such as vegetable oils and fats has been reviewed widely. Its advantages over petroleum diesel is safe, renewable, non-toxic, and biodegradable; it contains no sulphur; and it is a better lubricant. The utilization of fly ash as a suitable feedstock for use as heterogeneous catalyst for the transesterification would allow benefit of fly ash in an environmentally friendly way which would make the process more economically. In this research, solid fly ash prepared and treated using KOH to become an base catalyst. Then, transesterification process carried out by mixing *Jatropha curcas* oil, catalyst, and methanol to become methyl esters. This study showed that temperature affects on yield (%) of biodiesel production. Higher temperature increase the yield (%)**.**

Keyword : Biodiesel, Fly Ash, Transesterification, Jatropha curcas

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1. INTRODUCTION

Energy consumption is inevitable for human existence. There are various reasons for the search of an alternative fuel where can increasing demand for fossil fuels in all sectors of human life and that fossil-fuel resources are non-renewable.. Several alternatives such as wind, solar, hydro, nuclear, biofuel, and biodiesel have been suggested but all of them are still in the research and development stage.

Biodiesel or methyl ester is a fuel from vegetable oils that have properties similar with petroleum-based diesel. Biodiesel can be used either pure or blended with petrodiesel without changes on other machines that use it. The use of biodiesel as an energy source are demanding to be realized. Because, besides being a solution to the scarcity of fossil energy in the future, biodiesel also be renewable (renewable), can decompose (biodegradable), has the lubrication properties of the piston engine because it includes a group of non drying oil (non-drying oil), is able to reduce emissions carbon dioxide and the greenhouse effect. Biodiesel is also environmentally friendly because emissions gas is much better than petroleum-based diesel which is free of sulfur, the number of smoke (smoke number) lower, burn completely (clean burning), and does not produce toxins (Mardiah et al, 2006).

The satisfactory replacement of petroleum diesel with biodiesel is feasible only if it encounters two basic requirements: first is its easy availability and environmentally acceptability and second being economically reasonable. Thus, feedstocks for biodiesel production could be divided into different categories such as edible oils, non-edible oils, waste oils, animal fats, and algal lipids. (Helwani et al, 2013). There is a growing interest in using *Jatropha curcas* oil as the feedstock for biodiesel production because it is non-edible and thus does not compromise the edible oils, which are mainly used for food consumption. Non-edible oils are not suitable for human consumption because of the presence of toxic components. Further, *J. Curcas* seed has a high content of oil and the biodiesel produced has similar properties to that of petroleum-based diesel.

The problem beside that is Jatropha's oil which is difficult to find and doubt its purity and the production process of biodiesel both tools and materials that are not commercially yet available. This research is aimed to study the affect of fly ash catalyst loading to the transesterification yield and conducted by several factors and variables.

2. METHODS

There are four primary methods to make biodiesel: blending, microemulsion, pyrolysis and transesterification. The most commonly used method is the transesterification. Transesterification is the replacement of the alcohol groups of the ester by another alcohol in a process that resembles hydrolysis. But different with hydrolysis, the transesterification process are used alcohol, not water. Some types of alcohol used in the transesterification process is methanol, ethanol, propanol, butanol, and amyl alcohol. Methanol is more commonly used for the transesterification process because they are cheaper and easier to be recovered, although it is possible to use other types of alcohol. This reaction can be catalyzed by base (Maa and Hannab, 1999), acid or enzyme (Jothiramalingam and Wang, 2009). Base-catalyzed transesterification is much faster than acid, so widely used in commercial usage.

Solid fly ash-base catalysts were prepared and their catalytic performance in transesterification of *Jatropha curcas* oil was studied. In this study, fly ash loaded with KOH was assessed for the transesterification of *Jatropha curcas* oil with methanol to methyl esters.

The main material used in this experiment is *Jatropha curcas* oil and Fly Ash. *Jatropha curcas* oil took from Semanggi, Solo, Central Java and fly ash bought from PT. Petrokimia Gresik, East Java. For analysis and auxiliary materials to be used in this research are :

a. Methanol
b. Jatropha curcas oil
c. Fly ash as catalyst
d. KOH

The procedure of this research, first, fly ash preparation : We weighed fly ash and washed it using aquadest, dried it 110 ℃ for 3 hours using oven, then cooled it using desicator, screened 120 mesh, mixed fly ash and KOH 8M for 24 hour, screened and washed it with aquadest until pH 7, dried it 110 ℃ for 3 hours using oven, fly ash ready to use.

Second, Acid Value Test : Weighed oil samples 5 gram into a 250 ml erlenmeyer, added 50 ml methanol into the sample and dissolved, then added 3 drops of indicator phenolphthalein, boiled it for 30 minutes, titration with 0.1 N KOH standard solution which has standardized with $H_2C_2O_4$ until the color changed into pink.

$$\text{Acid Value} = \frac{mL\ KOH\ x\ N\ KOH\ x\ 56{,}1}{gram\ of\ oil}$$

Third, Transesterification Test : Prepared 100 grams of oil in beakerglass, then prepared certain catalyst and 41,5 ml of methanol in beakerglass too, stired and heated 40 ℃, then put in three neck flash, stired and heated at certain temperature, stop reaction when 90 minutes left, the results of the reaction moved into vacuum erlenmeyer to separate the solid catalyst, then moved into a separating funnel and let it separated between methyl esters and glycerol, glycerol in top layer and methyl ester di bottom layer, put methyl esters in rotary evaporator to removed methanol, createed analysis data of volume, mass and % yield of the results obtained.

Analysis of manufacturing methyl ester (biodiesel) and the characterization of methyl esters (biodiesel) produced is as follows :

a. The content of methyl esters (biodiesel) detected by *Gas Chromatography-Mass Spectrometry* (GC-MS)
b. The mass of methyl ester produced by weighing mass of methyl ester produced.
c. The amount of methyl ester produced by weighing volume of methyl ester produced
d. Percent of yield calculate by :

$$\%\ \text{yield} = \frac{mass\ of\ methyl\ ester\ (gram)}{mass\ of\ initial\ oil\ (gram)} \times 100\%$$

This research is aimed to study the affect of fly ash catalyst loading to the transesterification yield and conducted by several factors and variables :

a. Amount of catalyst (Percent of Fly Ash) = 2% ; 3% ; 4% ; 5% ; 6% ; 7% ; 8%
b. Temperature = 30 °C ; 45 °C ; 60 °C
c. Ratio of mole of *Jatropha curcas* oil and mole of methanol (1:9)
d. Reaction Time = 90 minutes

3. RESULT AND DISCUSSION

In the process of making biodiesel from *Jatropha curcas* oil using catalysts of fly ash with alkaline treatment, it was obtained relationship between variation of % transesterification catalysts and temperature.

3.1 Correlation Between Volume and % of Catalyst

Data volume of biodiesel results of the experiment are as follows (Table 1).

Table 1.Data of Volume Biodiesel in (ml)

% of Catalyst	Temperature (°C)		
	30°	45°	60°
2	55	55	58
3	50	53	57
4	49	51	54
5	46	54	56
6	42	43	59
7	44	43	44
8	40	48	55

It can be seen in Table 1 that in the treatment with 3% of catalyst the result of the volume of biodiesel at every increasing temperature also tends to increase. At a temperature of 30°C , 45°C , 60°C volume biodiesel are 50 ml and 57 ml respectively.

It can be seen that the temperature affects the yield (%) of biodiesel production. Higher temperature increase the yield (%). This is because with the increasing temperature will speed up the reaction so yield (%) of products is increasing. The increasing reaction temperature will increase kinetic energy of particle then will increase inter-particle collisions so that reaction go faster (Lestari, 2011).

In the process of producing biodiesel from *Jatropha curcas* oil using catalysts of fly ash with alkaline treatment, obtained relation between variation of % transesterification catalysts and temperature

3.2 Correlation Between Temperature and % of Catalyst

Data of mass of biodiesel results of the experiment are as follows (Table 2).

Table 2. Data of Mass of Biodiesel in (gram)

% of Catalyst	Temperature (°C)		
	30°	45°	60°
2	54,8226	55,078	58,2201
3	49,7728	52,7756	56,5963
4	49,1233	51,1378	54,3900
5	45,8322	54,1425	55,5411
6	41,5243	42,9182	58,8435
7	43,9415	43,0305	44,1819
8	40,4205	47,5814	55,2086

Table 2 showed the effect of temperature of reaction and amount of catalyst added. It is exhibited that temperature of reaction affect the resulted biodiesel mass. However the amount of catalyst does not influence significantly to the biodiesel mass. Treatment with 2% of catalyst show at a temperature of 30° mass biodiesel is 54,8226 gram, at temperature of 45° mass biodiesel is 55,078 gram, at temperature of 60° mass biodiesel is 58,2201 gram. It can be concluded that the volume of biodiesel is directly proportional to the mass of biodiesel.

3.3 Correlation between Catalyst and Yield Produced

In the process of making biodiesel from *Jatropha curcas* oil using catalysts of fly ash with alkaline treatment, obtained relation between % catalysts and yield of biodiesel. Data of correlation between catalyst and yield produced of the experiment are as follows :

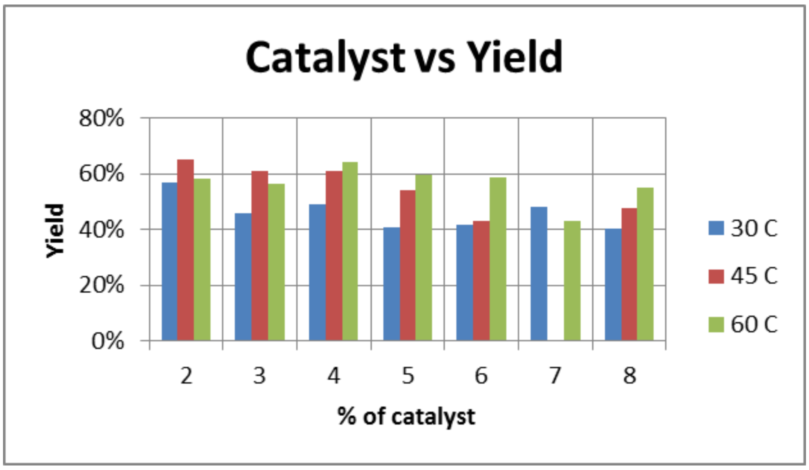

Figure 1. Correlation between Catalyst and Yield Produced

Figure 1 is relationship between yield at biodiesel and amount of catalyst. Percent yield of each increasing temperature is likely to increase. So the relationship between both of them to increasing of % catalyst which also tends to increase, despite decline in the 6% catalyst that caused the data tend to be fluctuating.

Based on the previous studies that the little amount of catalyst, makes yield to be higher. Though the increase of the catalyst will increase the reaction rate so that the purity of biodiesel produced will increase. That is because when amount of catalyst used is too high causing mixture between catalyst and reactant becomes thick (large viscosity) so that the impact on the decresing yield. When a catalyst is added much, slurry (a mixture of catalyst and the reactants) is too thick, causing problems in mixing and ultimately affect the yield value.

The effect of the amount of catalyst caused decreasing of % yield of biodiesel, by increasing the amount of catalyst will be more amount of biodiesel products are absorbed into the catalyst. This transesterification reaction using fly ash as a catalyst. Use of fly ash as a solid base catalyst has many advantages, such as high activity, low reaction conditions, a long lifetime of the catalyst and lower costs. This is because not washing the catalyst to regain biodiesel absorbed.

In the process of making biodiesel using fly ash base catalyst, fatty acid will react with the catalyst to form the soap through the saponification reaction, so the effectiveness of the catalyst will decrease and these conditions would also reduce yield ester and complicate separation process.

In the liquid base catalyst (KOH), the results of the transesterification process of biodiesel product, color is more yellow clear than the solid base catalyst, this is because the absence of a catalyst slurry of catalysts and reactants, and makes lower viscosity

3.4 Biodiesel Product Characterization

The following methyl ester analysis using Gas Chromatography - Mass Spectrometry (GC-MS). The analysis is to determine whether there is content of Fatty Acid Methyl Ester (FAME) to produce biodiesel. The content of biodiesel is show in Table 3.

Table 3. Identification and Product Composition Biodiesel (FAME) Liquid by GC-MS.

Peak	Component		Composition (%)
1	$C_{17}H_{32}O_2$	Methyl Palmitoleate	0,87
2	$C_{17}H_{34}O_2$	Methyl Palmitate	16,85
3	$C_{19}H_{34}O_2$	Methyl Linoleate	20,89
4	$C_{17}H_{36}O_2$	Methyl Oleate	51,56
5	$C_{19}H_{38}O_2$	Methyl Stearate	9,59
6	$C_{15}H_{26}O_2$	Methyl Pentadec	0,25

Table 3 show that the highest area reached the peak 4, while the retention time at that peak is 39.515 min. Complete data analysis results of GC-MS is shown in the attachment. Besides the quality biodiesel is not determined by the type of compounds contained in them, but from the value of density, viscosity, acid number, cetane number and the physical properties of other diesel fuels.

For comparation, this is results from another research to make biodiesel using fly ash but on sun flower oil.

Table 4. Product Composition Biodiesel (FAME) Liquid by GC-MS on Sunflower Oil

Peak	Component		Composition (%)
1	$C_{17}H_{32}O_2$	Methyl Linolenic	0,56

2	$C_{17}H_{34}O_2$	Methyl Palmitate	6,08
3	$C_{19}H_{34}O_2$	Methyl Linoleate	71,73
4	$C_{17}H_{36}O_2$	Methyl Oleate	16,93
5	$C_{19}H_{38}O_2$	Methyl Stearate	3,26

It can be seen and compared that Jatropha's oil has more component of fatty acid than sunflower's oil. In Jatropha, the higher component is methyl oleate but in sun flower's oil is methyl linoleate although that research used same catalyst and process.

4. CLOSING

The conclusion that can be drawn from the research that has been carried out as follow. Use of fly ash as a solid base catalyst has many advantages, such as high activity, low reaction conditions, a long lifetime of the catalyst and lower costs. The optimal temperature for the transesterification process of making biodiesel using Jatropha oil is 60°C. Temperature effect on yield (%) of biodiesel production. Higher temperature increase the yield (%). Lower amount of catalyst caused decreasing % yield of biodiesel, cause increasing the amount of catalyst will be more amount of biodiesel products are absorbed into the catalyst. The biggest component of FAME in this biodiesel is methyl oleate which is 51,56 %.

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